An Experimental Investigation of Tribological Characteristics of Environmentally Acceptable Lubricant Containing Ionic Liquid and Nanoparticles as Additives

A

Research Proposal (Synopsis) Submitted

(Doctor of Philosophy)

Under the supervision of

Prof. H. C. Garg

Department of Mech. Engg.

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Gitesh Kumar

190160090104

Pre-Ph.D

DEPARTMENT OF MECHANICAL ENGINEERING

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HISAR-125001 (HARYANA)

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1. INTRODUCTION

1.1 Background and Motivation

Lubricants are the material which is used to minimize friction and wear between two moving surfaces. Additionally, it prevents metal from corrosion, dissipates heat, clean away contaminants, and acts as seal to outside components [Maleque et al. 2003]. Since the ancient time of roman, oil extracted from plant and animal fat, lime, soap, and water being used as a lubricant. At that time natural resources were used as lubricants and life of peoples was environment friendly. But as time passed, animal or vegetable oil fails to meet the lubrication requirements of modern machinery and being forced to the utilization of mineral and synthetic lubricants. The first commercial petroleum well-being in Titusville, PA, USA, 1859 started the new petroleum oil industry. Nowadays, mostly available lubricants in the market are based on mineral oil because of their low cost and superior performance. The utilization of bio-based lubricants was completely declined by the end of the 20th century. Mineral oils are directly produced from petroleum or crude oil and synthetic oils are manufactured by the addition of some additives in mineral oil according to application. But it should be noted that both mineral and synthetic oils are not environment friendly. By the end of the 20th century, adverse effects on the environment due to toxicity and non decomposability of petroleum based and synthetic oils were realized [Tamada et al. 2012]. Dispersing and tossing of utilized petroleum based and synthetic oils are a threat to wildlife, fishes, and plants [Aluyor and Ori-jesu 2009]. Also, this spilling of oil is continuously damaging the physical and chemical properties of the sand day by day [Kayode et al. 2009]. Petroleum based lubricants cause environmental issues and these issues cannot be ignored anymore.

In recent years, awareness of the deteriorating environment has forced to reutilization of bio-based oil. Researchers are exploring the various form of bio-based lubricants. In developing countries, such as India, the environmentally acceptable lubricants are more widely regarded as renewable lubricants and alternative sources of lubricants in the future. Portugal is the first nation that mandated the use of environmentally friendly lubricants for the two-stroke engine. The selection of lubricants currently takes into account costs rather than efficiency and lastly environment. Legislative pressure and economic stimuli could drive the lubrication industry to eco-friendly lubricants. Many countries that have prohibited the utilization of dangerous lubricants such as Europe have been extremely restrictive its utilization in environmental applications since 2000 legislation. In recent years, the commercial interest in the use of bio-based, renewable, or plant-oil-based lubricants has grown considerably, owing to the criteria for lubricants and to tightening environmental standards. As a result, from 2016 to 2024, the worldwide demand for environmentally acceptable lubricant is expected to increase to 5.4 % annually [Tsagaraki et al. 2017]. The high biodegradability and superior tribological performance of vegetable oils have made it suitable alternatives for petroleum

lubricants. But Alone vegetable oils has its limitations as a lubricant [Campanella et al. 2010]. Future lubricants are likely to be environmentally acceptable although ensuring reasonable lubricating characteristics. Several large petroleum's and the chemical manufacturer have already patented bio-lubricants technology, such as ExxonMobil, Shell, Chevron, BASF and Dow.

1.2. Vegetable oils

Vegetable oil is the most suitable candidate for base lubricant concerning for environmentally acceptable lubricant. The most common crops for producing vegetable oil are canola oil, rapeseed oil, coconut oil, castor oil and soybean oil. Vegetable oils have serval unique properties and limitations when considering as a lubricant. Vegetable oils have better viscosity, excellent biodegradability and low toxicity, higher lubricity or ability to reduce friction, high flash point and they have the capability to work at extreme pressure and don't react with paints, seals and varnishes. On the positive side, vegetable oil has superior lubricity than mineral oil. Vegetable oils have higher viscosity index (150-250) than mineral oil (90-100). Vegetable oils have high flash point and fire point (around 326°C) than common mineral oil (200°C). Physicochemical properties of most common vegetable oils are given in table 1.

Table 1. Physicochemical properties of most common vegetable oils [Syahir et al. 2017]

Sr. No	Vegetable oil	Density @ 15°C (Kg/m^3)	Kinematic viscosity @ 40°C (mm^2/s)	Kinematic viscosity @ 100°C (mm^2/s)	Viscosity Index	Flash Point (K)	Pour Point (K)
1	Castor oil	0.950-0.970	220.6	19.72	220	523	246
2	Soybean oil	0.922-0.934	28.86	7.55	246	598	264
3	Rapeseed oil	0.910-0.917	45.60	10.07	180	513	261
4	Rice Bran oil	-	40.6	8.7	201	591	260
5	Sesame oil	-	27.33	6.3	193	589	268
6	Coconut oil	0.919-0.937	24.8	5.5	169	598	294
7	Corn oil	0.916	32.41	8.06	238	597	259
8	Sunflower oil	0.920-0.927	40.05	8.65	203	525	261

On the negative side, there are some limitations in vegetable oil, poor performance at both higher and low temperature, oxidation instability, unpleasant smell, filter clogging tendency, high pour point, and high CoF at elevated temperature. These limitations may be overcome by

uses of high oleic oils and suitable additives. More oleic acid, monounsaturated fat, and less polyunsaturated fats are the less oxidation-sensitive oils. Thus, oils with a high content of monounsaturated fatty acids have superior lubricity. Environmentally acceptable lubricant involves the majority of lubricant is biodegradable and contains a few percentages of additives.

1.3 Enhancement through additive

Additives are necessary to improve the existing properties of base oils. Commercial mineral oil can consist of 10-25 % additive depending on applications. Additives are expected to provides numerous features to base lubricants, such as viscosity index improvers, corrosion inhibitors, antioxidants, antiwear additives, and friction modifiers.

Nanoparticles

Material with any external dimension in the nanoscale size of one billionth (10^9) meter is nanomaterials. The mixture of nanomaterials and base lubricating oil is termed as nano-lubricant. The small size allows the nanoparticles to reach the contact region, resulting in a favourable lubrication effect, is the most important feature. In addition, the filters used in oil systems are thin enough to flow uninterruptedly. Nanoparticles are used as a lubricant mediator for many applications. Five advantages of nanoparticles being used as lubricant additives: (1) insoluble; (2) non-reactive to other lubricant additives; (3) high film-forming capability; (4) durable; and (5) non-volatile even at elevated temperature. Several researchers have confirmed that a specific type of nanoparticles is being used as antiwear, Extreme pressure, and friction modifier for many purposes.

Ionic liquids

Room temperature ionic liquid salts are the combination of organic cations (large) and anions (smaller) having melting temperature below 100˚C. It is predicted that up to a trillion (10^{18}) types of ionic liquids could be formulated with different cation and anion combinations. Ionic liquids are thermally stable compound and they have a large operating temperature up to 300˚C where they maintain their fluidity. Ionic liquids have given a lot of attention in Tribology over the last decade, because of their distinctive properties and capabilities such as low volatility, negligible vapour pressure, fire resisting, and superior chemical and thermal stability. Their peculiar properties appeal to the most diverse technological area such as chemical industries. The unique properties of ionic liquid differentiate them from conventional synthetic oil used as lubricants. Ionic liquids have melting point below 100˚C and most of them are available in liquid form at room temperature. Ionic liquid has zero vapour pressure even at elevated temperature. Ionic liquids are highly thermal stable greater than 350˚C. The viscosity of the most ionic liquids relatively high compared to conventional lubricants such as water. Ionic liquids are denser than organic solvent.

So, the ionic liquids are the suitable replacement of lubricant or lubricant additive for next-generation lubricants. In 2001, Ionic liquids were first time used as a neat lubricant. [Ye et al. 2001] The higher cost of ionic liquid limited its uses as neat lubricants, despite the circumstance that their enhancing tribological properties have been universally accepted. For circumstances where a large amount of lubricants, such as gearboxes and engines, is necessary, the replacement of petroleum lubricants with ionic liquid is not acceptable. Therefore, an alternative way is to combine the ionic liquid with lubricant. Imidazolium, phosphonium, pyridinium and ammonium cations are the most common class of IL used as lubricants. Most ionic liquids are formed of anions that contain halogen, which can be decayed into hazardous and corrosive substances. So, the halogen-free ionic liquids are gaining large interest in a tribological application. Many studies focus on the imidazolium cation paired with tetrafluoroborate (BF_4) and hexafluorophosphate (PF_6).

2. LITERATURE REVIEW

The tribological characteristics of various lubricants have been scanned for the purpose of researching the effect of ionic liquids and nanoparticles as additives. This section reviews the tribological investigation of ionic liquid as neat lubricant, ionic liquid and nanoparticles as additives in various base lubricants. Also, the synergistic effect of ionic liquids and nanoparticles as additives in various base lubricants has been reviewed.

2.1 Review of nanoparticles as an additive in lubricants

Arumugam and Sriram 2013 Compared the tribological behaviour of chemically modified rapeseed oil, with and without the inclusion of titanium dioxide (TiO_2) nanoparticles. The result showed that the inclusion of nanoparticles showed good reducing friction and antiwear properties. Xu et al. 2014 The tribological properties of soybean oil (SO) were studied with various form additives of molybdenum disulphide (MoS_2). MoS_2 hollow nanospheres enhance the tribological properties of SO. The tribochemical reactions between MoS_2 hollow nano-sphere, SO and friction content create a lubricating film made of the compounds MoO_3, Fe_2O_3 and carbon. Gu et al. 2014 evaluated the tribological characteristics of cerium-doped anatase nanophases composite (20 nm average diameter) as additives in rapeseed oil on a four-ball tribotester. Results showed that additives enhance the ability of rapeseed oil to minimize wear and friction particularly for 2.0 and 3.0 percent molar doped-in nanophases of cerium. Koshy et al. 2015 The tribological characteristics of coconut oil and mineral oils (500N base oil) with unmodified or surfactant-modified nanoparticles of molybdenum disulphide (MoS_2) were examined using a modified pin-on - disc tribometer and a four-ball tester. The optimal nanoparticle concentration of MoS_2 is measured at 0.53 and 0.58 respectively, in coconut oil and 500N basis oil.

Gulzar et al. 2015 The anti-wear (AW) and extreme pressure (EP) properties of chemically modified palm oil (CMPO) with CuO and MoS_2 nanoparticles were evaluated experimentally with four-ball and sliding wear tests. The MoS_2 nanoparticles had excellent AW / EP properties than the CuO nanoparticles. Kashyap and Harsha 2016 examined the tribological characteristics of rapeseed oil with CuO and CeO_2 nanoparticles (less than 50 nm) ranging from 0.1 to 1 wt% concentration using a four-ball tester. Results showed that chemically processed rapeseed oil has strong anti-wear and friction-reducing properties compared with raw rapeseed oil. Reeves et al. 2017 examined the tribological performance of various vegetable oils with hBN (hexagonal boron nitride) as an additive by using the pin-on-disk tester. Avocado oil showed the highest tribological efficiency relative to other vegetable oils. lubricant mixtures had a friction reduction of 8 to 64 % for micron-sized to nano-sized particles, respectively. Wear decreased even for particulate matter, which varies from micron to nano, with a decrease of 13 to 70 %. Gupta and Harsha 2018[a] examined the extreme pressure, anti-wear, and anti-friction efficiency of castor oil with CeO_2 and polytetrfluroethylene

nanoparticles with four different concentrations in the range of 0.1 to 1 w/v% of using a four-ball tester. For anti-wear tests, the optimal amount of CeO_2 and polytetrafluroethylene additives was reported to be reduced by respectively 37.4 and 35.3%. Gupta and Harsha 2018[b] examined the anti-friction, antiwear, and extreme pressure characteristics of unformulated and formulated castor oil with modified CuO nanoparticles with varying concentrations in the range of 0.1 to 1.0 w/v% using a four ball tester. For the wear test, wear scar diameter decreases were the highest at 28.3% and 22.2%. But the friction coefficient was reduced by 34.6 and 17.3 % at an optimal amount of nanoparticles in castor oil and paraffin oil, respectively.

Shafi et al. 2018 observed that Cu nanoparticles in avocado oil led to a reduction in friction and wear. At the concentration of 1 wt.%, the coefficient of friction was observed minimum, whereas the minimum specific wear rate was at 0.5 wt%. Bhaumik et al. 2018 The higher concentrations of ZnO Nano friction modifiers have seen a growing improvement in viscosity. In the case of castor oil, the friction coefficient is observed to be lower than the mineral oil, but above the optimum concentration 0.1wt % of ZnO, the friction coefficient does not decrease. Keshtiban et al. 2019 examined the friction coefficient of SiO_2 nanoparticles based lubricants with different weight concentrations by standard compression test. Results revealed that SiO_2 nanoparticles in base lubricants significantly improved the lubrication efficiency and reduced considerably both the coefficient of friction and the surface roughness. Kerni et al. 2019 carried out the epoxidation of olive oil to enhance its oxidation stability and the epoxidized oil showed better lubricating performance compared to virgin olive oil. An additional increase was also achieved in the addition of nanoparticles, in which a minimum friction coefficient was observed at 0.5 wt. % concentration.

Shafi et al. 2019 examined the tribological performance hazelnut oil with copper (Cu) nanoparticles in 0.5 and 1 Wt.% concentrations using a pin-on-disc tribometer. An improvement in friction coefficients of about 80 percent and a particular in specific wear rate of around 99 percent was observed. The lubricant showed improved results with 0.5 Wt.% Cu nano-particles. Kumar et al. 2019 examined the tribological properties at four different concentrations of 0.04, 0.05, 0.1 and 0.2 wt.% of copper oxide nanoparticles and a constant 1 wt. % of ZDDP with refined soybean oil using c pin on disc tribotester. In comparison to base oil, the inclusion of ZDDP and CuO nanoparticles in RSBO can minimize friction and wear. The optimum nanoparticle concentration of CuO in RSBO is 0.05% wt. Cortes et al. 2020 investigated the rheological and tribological characteristics of SiO_2 and TiO_2 nanoparticles in sunflower oil as additives in various concentrations. Results indicated that a reduction in friction coefficient of 77.7% and 93.7% was observed compared to base sunflower oil respectively. Furthermore the volume losses were decreased by 74.1% and 70.1% with the inclusion of SiO_2 and TiO_2 nanoparticles.

2.2 Review of ionic liquids as lubricants

Ye et al. 2001 introduced ionic liquids [HMIM][BF_4], [EMIM][BF_4], fluorine-containing lubricants phosphazene and perfluoropolyether first time as lubricants. Better friction reduction properties, antiwear performance and high load carrying capacity were observed for the selected ionic liquids. Liu et al. 2002 investigated the tribological performance of alkylimidazolium tetrafluoroborate with an Optimol SRV oscillating friction and wear tester in air and a CZM vacuum friction tester with steel and steel contact. Results demonstrate that ionic liquid had superior tribological performance to phosphazene (X-2P) and perfluoropolyether, both in air and in vacuum. The surface protective film is mainly composed of FeF_2 and B_2O_3 had reduced the friction and wear. Iglesias et al. 2004 investigated the friction and wear characteristics of the combination of n-dodecylammonium chloride, 4,4-dibutylazobenzene and cholesteryl linoleate with 1 wt.% of liquid crystalline additives.. The lowest friction coefficient was observed for 4,4-dibutylazobenzene and lowest wear volume was observed for n-dodecylammonium.

Wang et al. 2004 prepared and examined the tribological properties of alkylimidazolium ionic liquid lubricants as steel and steel contact lubricants on a wear tester and optimol SRV friction. The synthetic ionic liquids have excellent tribological properties and are comparable to the traditional paraffin lubricant, which contains 2 wt. % of zinc dialkyldithiophosphate, for a load-carrying capability. Doerr et al. 2005 explored the tribological properties of six different ionic liquids by using an optimol SRV oscillating friction and wear tester and compared with reference lubricants in steel and steel contacts. Two types of results: low friction, higher wear and higher friction, low wear as concerning reference lubricants. Suzuki et al. 2007 investigated the tribological and rheological characteristic of [HMIM][BF_4], and [HMIM][PF_6] under high vacuum conditions. In a high vacuum, Ionic liquids had high thermal characteristics of viscosity, high thermal stability, and low friction and low wear. Qu et al. 2009 investigated the sliding friction and wear properties of AISI 52100 steel and aluminum alloy lubricated with ionic liquid steel alloys at room and elevated temperature. The lubricating performance one ammonium-based [C_8H_{17}]3NHTf_2N and one imidazolium-based C10mimTf_2N, had better tribological performance compared to conventional fully-formulated engine oil.

Shah et al. 2011 synthesised, characterised and evaluated the tribological properties of hydrophobic and hydrolytically stable orthophoric ionic liquid (HF-BILs) at room temperature. These HF-BILs have significantly improved antiwear and friction reducing properties for steel-Aluminum contacts than that of the fully formulated (15W-50 grade) engine oil under boundary lubrication conditions. Jiang et al. 2013 prepared and examined the tribological properties of the phosphate ionic liquid on the Optical SRV oscillating friction at different loads and speeds. An extraordinary anti-wear capacity in comparison to steel/Al2024 in varying

loads and frequencies compared with traditional ionic liquids. Garcia et al. 2014 evaluated the tribological properties of ionic liquids [(NEMM)MOE][FAP], [BMP][FAP] and [BMP][NTf_2] as a neat lubricant for steel-steel contact using a HFRR tribometer and MTM tribometer at 40 and 100ºC. The [BMP][NTf_2] showed the lowest friction coefficient and wear due to its lowest viscosity. López Sánchez et al. 2014 evaluated ionic liquids [C_4C_1Pyrr][Tf_2N], and [$C_4C_1C_1$Im][Tf_2N], as appropriate lubricants for contacts with steel and steel using a reciprocating tribometer ball-on-plate under 8, 18 and 30 N loads at room temperature The findings were compared with a semi-synthetic oil formulated. Applying [$C_4C_1C_1$Im][Tf_2N], regardless of load, the best tribological action has been achieved. Kawada et al. 2014 elucidate the tribological properties of ionic liquids [EMIM][DCN], [EMIM][TCB], and [BMPL][TCB] on a sliding surface. Results revealed that the lowest coefficient of friction [BMPL][TCB] and the lower coefficient of frolic [EMIM][DCN] where determined to be [EMIM][TCB].

Otero et al. 2014 evaluated tribological behavior of the sliding pair AISI420 and 100Cr6 with two neat ionic liquids lubricants under loads 14, 18, 22, and 26 N. The ionic liquid [$P_{6,6,6,14}$][$(C_2F_5)_3PF_3$] showed better friction reduction and low wear volume properties than [$C_4C_1C_1$Im][$(C_2F_5)_3PF_3$]. Fan et al. 2015 investigated the tribological properties of halogen-free ionic liquid lubricants, 1-alkyl-3-methylimidazolium saccharinates and acesulfamates. These ionic liquids showed good lubricanting properties for both steel/steel and steel/copper friction pairs. Totolin et al. 2017 investigated the thermo-oxidative stability and tribological properties of halogen free ionic liquid using SRV tribotester. Phosphonium phosphate and a conventional friction modifier led to improved antiwear properties of the disc surface supported by the presence of iron phosphide and phosphate tribofilm. Fan et al. 2018 designed and synthesized by combining quaternary ammonium cation with a range of bio based fatty acid anion. From results it is revealed that this ionic liquid showed good and universal lubricants by steel and steel contact. Lubrication mechanism has demonstrated that the fatty acid antioxidant's polarity makes it possible to slide solid and efficient physical adsorption and tribochemical films.

Zhang et al. 2018 investigated the tribological properties of imidazolium based ionic liquid [$EMImBF_4$], [$BMImBF_4$], [$HMImBF_4$] and [$MOImBF_4$] of friction pairs steel-aluminium, steel-copper and steel-Si_3N_4 ceramic using ball-on-block reciprocating UMT-2MT tribometer. Results demonstrate that the lubrication effect is strengthened by increasing the alkyl chain length of the cations. The Ionic Liquids are strong tribology and hold high potential for both room temperatures and high temperatures on steel / copper, steel / aluminium, and ceramic pairs. Okubo et al. 2018 investigated the tribological performance of two different types of halogen-free ionic liquids: [BMIM][DCN] and [BMIM][TCC] with various contacts by using a reciprocating cylinder-on-disk tribotester. The friction test results showed a coefficient of friction ultrafree of 0.018-0.03, which was lubricated in ta-C – ta-C tribopair with [BMIM][DCN] or [BMIM][TCC]. Zhu et al. 2019 investigated phosphonium ionic liquids

tribological properties as standalone lubricants for stainless steel ball-disk configuration at room temperature and high temperature (100°C). The Ionic liquids were found to be noncorrosive and extremely thermal stable and had a greater wear and frozen wear and friction decrease than PAO10 synthetic lube oil and hexafluorophosphate (L-P106) commonly used in IL 1-methyl3-hexylimidazolium. Sheng et al. 2019 evaluated friction and wear pattern between steel surface was conducted in ball-on - disc sliding friction tests for 1-octyl-3-methylimidazolium ionic liquide Lactate (L-L108). The friction coefficient of L-108 was calculated at a 5 N load with a rotational speed of 200 rpm as 0.023 and is roughly 1/3 and 1/5 as L-P108 (0.068) and the SAE 10W-40 motor oil values (0.107), respectively.

2.3 Review of ionic liquid as an additive in lubricants

Battez et al. 2009 investigated the tribological and rheological performance of two room-temperature ionic liquids, [HMIM][BF_4] and [HMIM][PF_6] with 1 wt.% additive in mineral hydrocracking oil for steel and steel contacts. Results revealed that by addition of ionic liquids of base fluid its viscosity was increased and friction and wear characteristic were decreased. Qu et al. 2012 investigated trihexyltetradecylphosphonium bis(2-ethylhexyl) phosphate as antiwear lubricant additive in PAO and 5w30 oil. The ionic liquid is thermally stable up to 347 °C, showed no corrosion on cast iron, and has excellent wettability on solid surfaces. the friction coefficient was reduced by 60% and the wear rate by 3 orders of magnitude. Yu et al. 2012 investigated the tribological properties on plint TE77 high-frequency tribometer for synthetic polyalphaolephine (PAO) 4cSt, SAE 10W and 10W-30 engine oil with tetradecyl trihexyl phosphonium bromide, bis(2-ethylhexyl) hydrogen phosphate, and sodium hydroxide, Bis(2,4,4-trimethylpentyl) phosphinic acid as additive. Results demonstrated significant reductions in friction and usage by applying a small amount of PP-IL to the base oil, e.g. , 5 wt %. Thermally more stable are PP-ILs relative to typical lubricants.

Totolin et al. 2013 evaluated the tribological properties phosphate based ionic liquid with an SRV tribometer using an oscillating steel and steel contact with ball-on-disc configuration. The addition of imidazolium borane ionic liquids significantly improved the tribological properties than a halogen ionic liquid whwn used as standalone and additive to lubricant. The formation of a phosphate based tribofilm confirmed by XPS analyses of the wear scars had reduced the friction and wear remarkably. Khemchandani et al. 2014 investigated the tribological properties of biocompatible ionic liquid, tributyl(methyl)phosphonium diphenylphosphate and P_{1444}DPP (IL1) and compared to amine phosphate using Optimol SRV oscillating tribometer and a pin-on-disc tribometer. Results revealed that IL1 has a better film-forming ability and excellent antiwear performance than amine phosphate. Also, the tribofilm layer formed on steel disc was observed using atomic force microscopy (AFM). Anand et al. 2015 investigated the tribological characteristics of trihexyltetradecyl phosphonium bis(2,4,4-trimethylpentyl) phosphinate and trihexyltetradecyl phosphonium bis(2-ethylhexyl) phosphate, as lubricating oil additives in the fully formulated diesel engine lubricants mineral oil, 15W40

and used oil using High Frequency reciprocating tribometer (Plint TE77). Results showed that the friction and wear efficiency of the used lubricant is increased by adding both ILs. However, new (fresh) and in-service lubricant samples were shown to increase wear. Grace et al. 2015 investigated the tribological behavior of two phosphonium-based ionic liquids containing 1, 2.5, and 5 wt% as additives in coffee bean oil in steel-steel contact using a block-on-flat reciprocating tribometer. The results of the test were compared with fully-formulated and commercially available lubricant. Results showed that applying ionic liquids to coffee bean oil reduces the steel disc wear volume and wear values are equal to that of the commonly available lubricant. Shanhua et al. 2016 investigated the tribological properties ionic liquid [HMIM][BF_4] as additive in percentages 0.6 wt.%, 1 wt.%, 2 wt.%, and 3 wt.%. with castor oil using a four-ball test rig. Results revealed that minimum wear scar diameter (WSD) occurs at 1 wt.% ionic liquid.

Nagendramma et al. 2017 investigated the tribo-performance of aspartic acid and glutamic acid derived ionic liquids polyol ester based bio-lubricant on a four-ball tribo-tester. The findings demonstrate that the polyol ester-IL forms a compatible blend and reduces friction and wear. The analysis carried out has shown that the ILs have strong potential for biodegradable lubricants to be used as additives. Zhu et al. 2017 synthesized three oil miscible ionic liquid tetradecyltrihexylphosphonium stearate (TTP-ST), tetradecyltrihexylphosphonium oleate (TTP-OL), tetradecyltrihexyl-phosphonium O,O'-diethyldithiophosphate (TTP-DDP). Even the friction-reduction and anti-wear effects were evaluated using Optimol SRV-IV oscillating reciprocal friction and wear tester in ILs as a lubricating additive in pentaerythritol oleate (PETO) and trimethylolpropane trioleate (TMPTO). The findings revealed that the lubrication properties of TTP-DDP were significantly better than those that tricresyl phosphate (TCP) by performing very well at 200°C. Cigno et al. 2017 investigated the tribological properties using the pin-on-disc tribometer of the two separate phosphonium-based ionic liquids as bio-oil additives. Find that the presence of ionic liquid additives significantly decreases the wear volume loss, particularly at the higher velocity observed, indicating a possible improvement in operation. Reeves et al. 2018 investigated the tribological functionality of ionic liquids as additives in avocado oil. Results showed that a more percentage combination of RTILs would increase tribological efficiency on a linear basis. The friction coefficient (COF) could be decreased by up to 68,88% and the wear volume could be decreased, depending on the form and percentage of RTIL with avocado oil. Sani et al. 2018 investigated the lubrication performance of two oil-miscible ionic liquids; [P_{66614}][Phosphinate] (PIL) and [N_{1888}][NTf_2] (AIL) at 1%, 5%, and 10% weight concentration.as additive in the modified Jatropha oil-based (MJO) lubricant as potential vegetable-based metalworking fluids. MJO+AIL10% and MJO+PIL1% lubricants have equivalent lubrication performance to that another lubricant. Corrosion inhibition has been improved, friction has been reduced superiorly, the area has been smaller used, the surface has

become excellent finished and the torque has increased. Zheng et al. 2018 investigated the tribological properties of phosphite binds to aliphatic acid as anion and triethylbenzylammonium serve as a cation, as lubricant additives in mineral oil (5CST) and refined vegetable oil (RSO). Results revealed that the antiwear, friction reduction and intense pressure characteristics of 5CST and RSO could be improved by these ionic liquids. Reeves et al. 2019 investigated both bio and nonbio-based ILs to understand their tribological performance. Among the ILs studied, the better tribological properties were found to be $P_{666,14}$Saccharinate, $P_{666,14}$Salicyate and $P_{666,14}$Benzoate. Huang et al. 2019 synthesized the two kinds of oil-soluble ionic liquids with the same cation but different anions. Then used as additive with different mass concentration into PAO10. The results revealed that for steel and steel contacts the mixtures respond better than standalone PAO10.

Jiang et al. 2020 synthesized an oil soluble multifunctional protic ionic liquid and investigated its tribological and antioxidant properties in polyalphaolefin (PAO4). The results showed that for PAO4, frictions and wear were reduced considerably by ionic liquid. The PAO4 blending with the IL produces a 555 min induced oxidation time, 8.2 and 3.5 times the pure PAO4. Khan et al. 2020 synthesized four ionic liquids and used as additives to the mineral or synthesic oil base, consisting of tetrabutyl and tetraoctyl-based composition of the quaternary. The cation ionic liquids of tetraalkylphosphonium displayed greater thermal stability, while the cation-based analog of tettraalkylammonium exhibited higher viscosity. The viscosity index is improved by these ionic liquids as an additive to PEG 200 and SN 150 lube base oils. The ideal PEG 200 oil dosage of 1.5 w/v of lauryl sulfate-based ionic liquids strengthened tribological characteristics dramatically decreased friction by 28% and wear by 91%. Syahir et al. 2020 investigated the tribological performance of trihexyl(tetradecyl)phosphonium bis(2,4,4-trimethylpentyl) phosphinate, trihexyl(tetradecyl) phosphonium decanoate, and 1-butyl-3-methylimidazolium tetrafluoroborate as additive in polyalphaolefin (PAO8) and bio-based oil trimethylolpropane trioleate (TMPTO using four-ball and high-frequency reciprocating rig (HFRR) tribotesters. Results revealed that tribological performance were improved of both PAO8 and TMPTO at an optimum concentration of 1wt%.

2.4 Review of synergistic effect of ionic liquid and nanoparticles in lubricants

Wang et al. 2011 developed and investigated the tribological characteristics of nanofluids Gold/1-butyl-3-methylmethyl-hexafluorophosphate (Au/[Bmim][PF_6]) with various stabilizing agents. The nanofluids with a high stability demonstrated significantly greater friction reduction and wear reduction relative to pure [Bmim][PF_6] and nanofluids with low stability. the friction coefficients and wear volumes of the CTABr under the 800N are 13.8 and 45.4 percent lower than those of the pure [Bmim][PF_6] at low volumetric concentrations. Kheireddin et al. 2013 investigated the tribological properties of the mixture of SiO_2 nanoparticles and 1-butyl-3-methylimidazolium (trifluoromethysulfony)imide ionic liquid for

a tribo-pair of stainless steel ball and a steel surface. Results elucidate that the optimum NP concentrations of 0.05 wt %, the friction coefficient was approximately 35 % lower than at higher nanoparticles concentrations of 43 wt % and 25% lower at low NP concentrations of 0.01 wt%. Yegin et al. 2017 investigated the tribological and rheological properties of octadecyltrichlorosilane (OTS) functionalized silica nanoparticles (NPs) in 1-butyl-3-methylimidazolium (trifluoromethysulfony)imide. The existence of functionalized ionic liquid nanoparticles in the ionic liquid at optimum concentration lowered the friction coefficient by 37% compared to ionic liquid and 17% compared to ionic liquids with silica nanoparticles. Li et al. 2018 examined the synergistic effects of polyethylene glycol (PEG) base oil with 2-Mercaptobenzothiazoline and Mo nanoparticles. With single additives these hybrid additives perform better neat PEG oil and PEG. The hybrid additives in the PEG oil have excellent friction and anti-wear efficiency only at 100°C, but they are not apparent at 20°C, thus requiring high temperature to cause synergistic effects and decrease friction and wear. Sabarinath et al. 2019 investigated the synergic effect of 1-butyl-3- methylimidazolium tetrafluoroborate and 1-hexyl-3-methylimidazolium tetrafluoroborate ionic liquid with SiO_2 nanoparticles as hybrid additives in sesame oil. In combination with the SESO IL2 additives OA-SiO2 nanoparticles have superior tribological features as OA-SiO2 in combination with the SESO IL1 additives. With the inclusion of the chosen additives, SESO's rheological and corrosive properties did not improve substantially. Nasser et al. 2020 investigated the synergistic effect of hexagonal boron nitride nanoparticles (at 0.1 wt.%) and three phosphonium based ionic liquid in a polyalphaolefin PAO 32 lubricants. Results revealed that nanodispersion remains stable up to 240 days. Also, hybrid additives improve their tribological properties rather than being used separately. Raman spectroscopy of worn out surfaces also confirmed the synergistic effect and showed the excellent tribological characteristic of (PAO 32 + Trihexyltetradecylphosphonium bis(2-ethylhexyl) phosphate + h-BN). Upendra and Vasu 2020 investigated the tribological properties of ionic liquid (IL) trihexyltetra-decylphosphonium bis(2,4,4-trimethylpentyl) phosphinate along with Al_2O_3, CuO, and SiO_2 nanoparticles (NPs) as a lubricant additive in a group 1 mineral base oil. Excellent synergies among IL, Al_2O_3 and CuO nanoparticles were found in the improvement of wear and extreme pressures. Friction decreased by 19% and 24%, compared to 32% and 36% respectively for Al_2O_3 and CuO hybrid nano lubricants. Han et al. 2020 investigated the friction, wear and extreme pressure behavior were studied of 0, 0.5, 1 and 2 wt percent diamond, and nanoparticles of zinc oxide (ZnO) suspended with a phosphonium ionic liquid stable base oil. The findings also shown the best anti-friction action in the base stock, bonded with the disk surface, but followed by increased wear.

2.5 Literature Gap

A state-of-the-art of the available literature concerning the ionic liquids as neat lubricants, nanoparticles and ionic liquids as additives in various base lubricants has been reviewed. From the last two decades, metal oxide nanoparticles are being extensively used as lubricant additives

in various base oils, and have gained much research attention due to their capability to enhance the tribological properties. After the revelation of ionic liquid in 2001, it has been widely researched as a neat lubricant or as an additive in vegetable, mineral, and synthetic oils. Various researchers have confirmed in their study that ionic liquids as neat lubricants have excellent tribological properties. However, their cost currently restricts the use of ionic liquid as a standalone lubricant and a possible alternative is to combine ionic liquid and lubricants. Ionic liquids have the potential to improve the tribological properties of base oils and can be used as additives for lubricating oils. Based on the available literature and to the best of my knowledge, the combined influence of halogen free ionic liquid and nanoparticles as additives in vegetable oil has not been investigated yet.

2.6 Problem identification

The problem identified for this research work is the an experimental investigation of tribological characteristics of environmentally acceptable lubricant containing ionic liquid and nanoparticles as additives.

3. OBJECTIVES OF RESEARCH WORK

Knowing the fact that environmental deterioration by continuously uses of mineral oils has promoted the development of vegetable oil based lubricants. Using vegetable oil as base lubricant is the first necessary step for the development of environmentally acceptable lubricant. Further, the additives like ionic liquid and nanoparticles are added in the vegetable oil to enhance the rheological and tribological properties of the lubricant. The main aim of the present research work is to develop the environmentally acceptable lubricant.

Broadly, this research work aims to achieve the following objectives.

- To formulate vegetable oil-based environmentally acceptable lubricant with the addition of ionic liquid and nanoparticles as additives.
- To investigate the rheological characteristics of the formulated environmentally acceptable lubricant.
- To investigate the tribological characteristics of the formulated environmentally acceptable lubricant.
- Compare the characteristics of formulated environmentally acceptable lubricant with conventional engine oil.

4. METHODOLOGY

In order to achieve the above objectives, the following methodology will be used.

- Selection of suitable vegetable oils, nanoparticles and ionic liquid for the formulation of environmentally acceptable lubricant.
- Analysis of dispersion stability and rheological characteristics of formulated vegetable oil-based lubricants with nanoparticles and ionic liquid additives by using UV-vis spectroscopy and rheometer respectively.
- Analysis of tribological characteristics of formulated vegetable oil-based lubricants containing nanoparticles and ionic liquids additives by conducting experiments on a ball on disc tribotester and a four ball tester.

The methodology will be carried out in the following steps:

The first step to achieve the objectives is to narrow down the literature for the selection of vegetable oils, nanoparticles and Ionic Liquid. Vegetable oil will be selected on the basis of physicochemical properties such as low viscosity and high viscosity index, low volatility and high oxidation stability.

The second step is to formulate the vegetable oil based lubricants by adopting efficient preparation techniques, and then dispersion stability of nanoparticles and ionic liquid additives in vegetable oil will be carried out. Further, the Rheological properties of formulated vegetable oil-based lubricant containing nanoparticles and ionic liquid will be obtained using a rheometer.

In the third step, the friction and wear characteristics of formulated vegetable oil-based lubricant containing nanoparticles and ionic liquid as additives will be evaluated by conducting the experiments on a ball on disc tribotester and a four ball tester. After that, the experimental results of formulated environmentally acceptable lubricants and conventional engine oil will be compared.

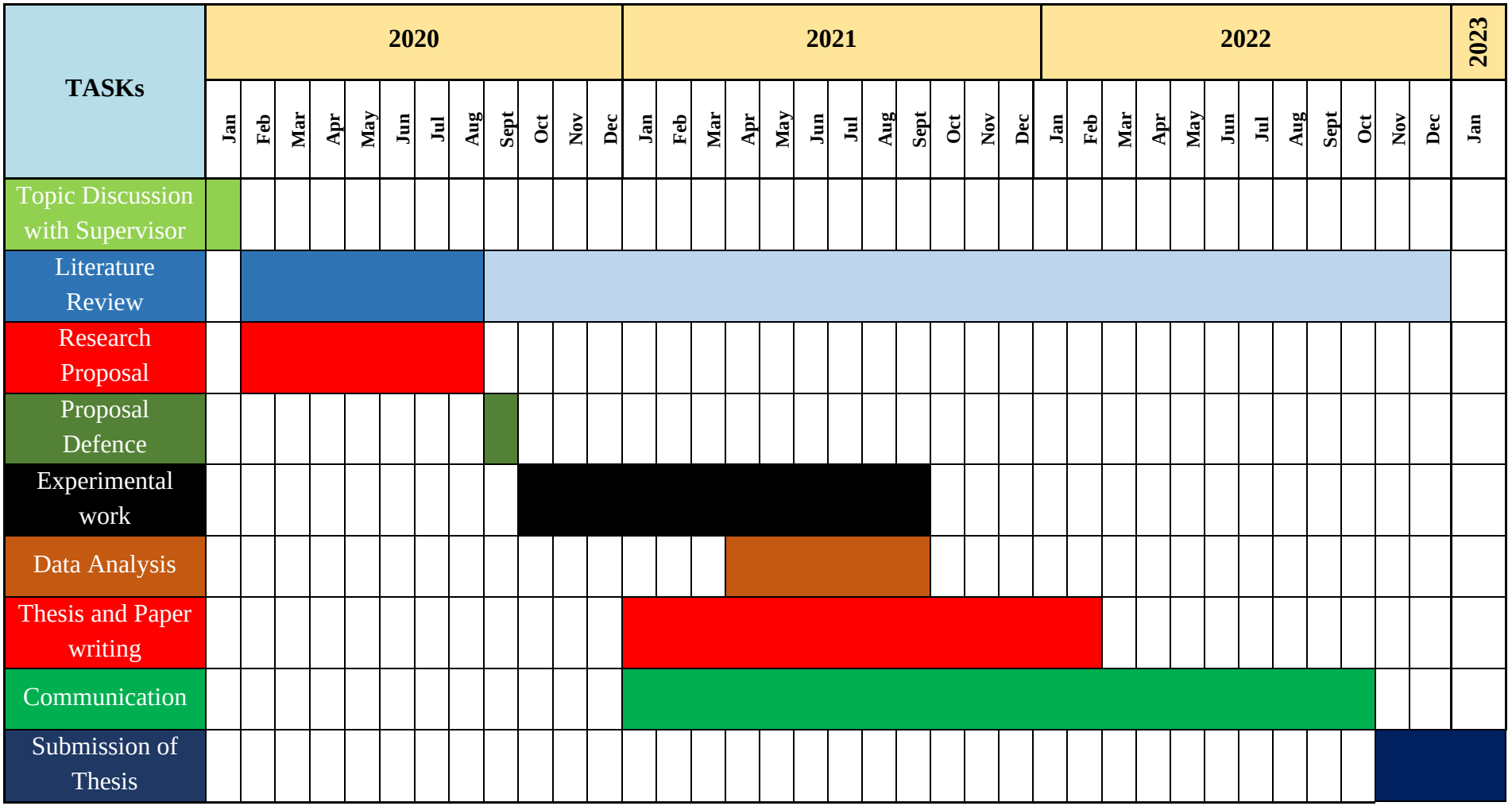

Figure 1 Ph.D Gantt chart

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